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Miniaturized, highly sensitive single-chip multichannel quartz-crystal microbalance

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A miniaturized highly sensitive single-chip multichannel quartz-crystal microbalance prepared by deep reactive ion etching is presented. In the present work, quartz resonators in a single-chip with the diameters in the range 0.05–1.0 mm and thicknesses in the range 18–82 μm were fabricated. The conductance measurements carried out on the resonators showed that the Q factor is inversely proportional to resonator thickness. The Q-factor value as high as ~30 000 has been observed in case of a 94 MHz resonator whose diameter is 1 mm and the thickness 17.8 μm. The Q factor of a resonator of very small diameter (0.1 mm) reached the value 5700. [DOI: 10.1063/1.1532750]

Quartz-crystal microbalance (QCM) sensors are commonly used for *in situ* deposition control of thin films. The operating principle of these QCM sensors is based on the fact that the change in resonant frequency of a vibrating quartz-crystal resonator is proportional to the mass of the deposited film.[1] In addition to thin-film deposition control, these devices are also employed for other applications such as gas phase detection,[2] immunosensing,[3,4] and deoxyribonucleic acid sensing.[5,6]

Generally, a conventional QCM consists of a circularly shaped quartz crystal of a diameter larger than 1 mm with electrodes deposited on either side of it. According to Shockley *et al.*,[7] the Q factor saturates to a maximum value when the ratio of the separation between the electrode and the boundary of the resonator to the thickness of the resonator is larger than 15. For sensor applications, AT-cut high-frequency fundamental quartz resonators with ''inverted mesa'' structure prepared mainly by a wet etching process have been reported.[8] However, there is an increasing interest to utilize a miniaturized single-chip multichannel QCM possessing high sensitivity for a multianalysis system.[9–11] In this letter, we report the fabrication and characterization of high Q-factor miniaturized single-chip multichannel QCM sensors.

In our design for the multichannel QCM sensors, we have maintained the separation of 1 mm between the circular resonators to reduce the effective interaction between them. The array of circularly shaped resonators with diameters in the range 0.05–1 mm were arranged on a rectangular quartz plate with the dimension of 12 mm×10 mm and thickness of 0.1 mm. Considering the possible application of the device in hard corrosive chemical media, platinum is chosen as the material for electrodes. In our design, the diameters of the electrodes are as same as diameters of the resonator cavities.

In order to prepare the multichannel QCM sensors, an AT-cut quartz crystal polished on both sides was chosen. To make quartz resonators of different thicknesses, we adopted the inductively coupled plasma reactive ion etching (RIE) technique. This technique offers the advantage of a good anisotropic etching ability. In this process, SF_6 and Xe gases have been used as etchants. The etching process pressure was maintained at 1 mTorr. The rf power used is 100 W with a

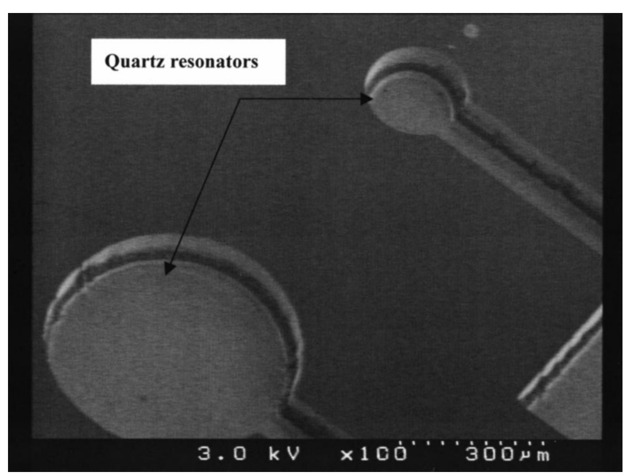

FIG. 1. SEM image of the quartz resonators.

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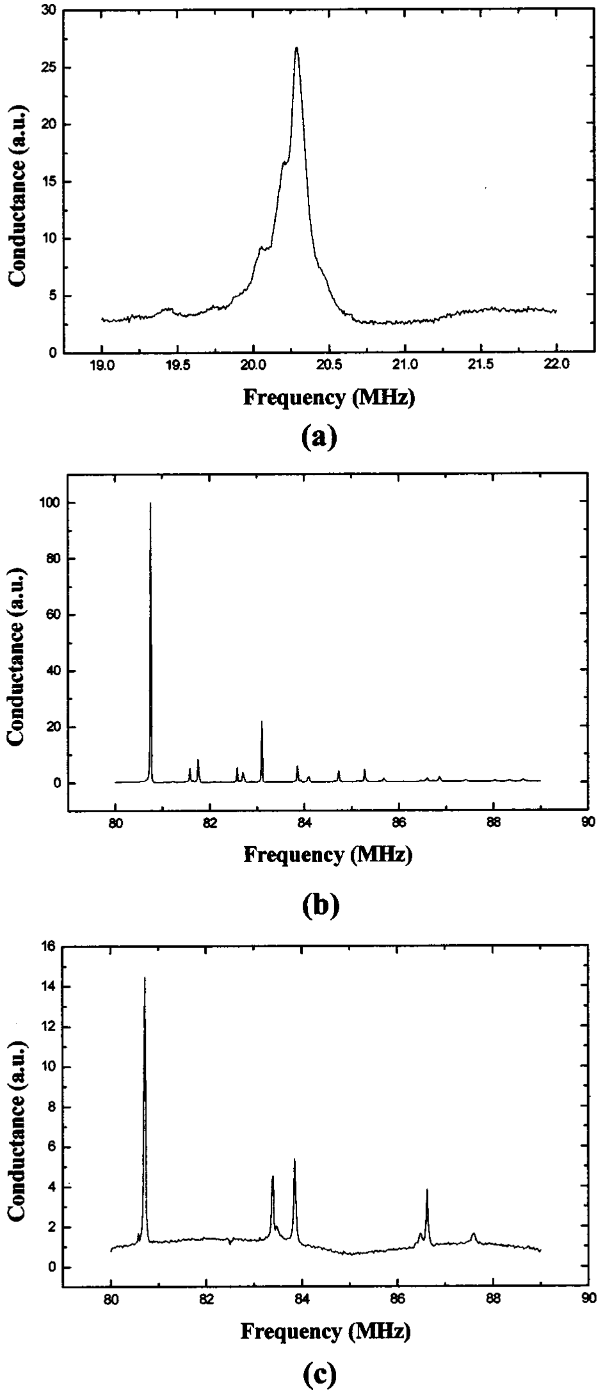

FIG. 2. Conductance characteristics of quartz resonators with (a) thickness ~82.3 μm and diameter 1 mm, (b) thickness ~ 20.7 μm and diameter 1 mm, and (c) thickness ~ 20.7 μm and diameter 0.1 mm.

self-bias of −340 V. The cathode temperature was maintained at 20 °C by circulating a coolant. The etch rate of the quartz sample was found to be 0.4 μm/min. This was calculated by measuring the total etched depth using a surface profiler. After the etching process, platinum/titanium electrodes with a thickness of 100/40 nm were deposited on the resonator using an electron-beam evaporation technique and lift-off process. The details of the procedure followed are given elsewhere.[10]

Figure 1 shows the scanning electron microscope (SEM) image of the fabricated quartz resonators. It is important to note that the surface roughness very much influences the

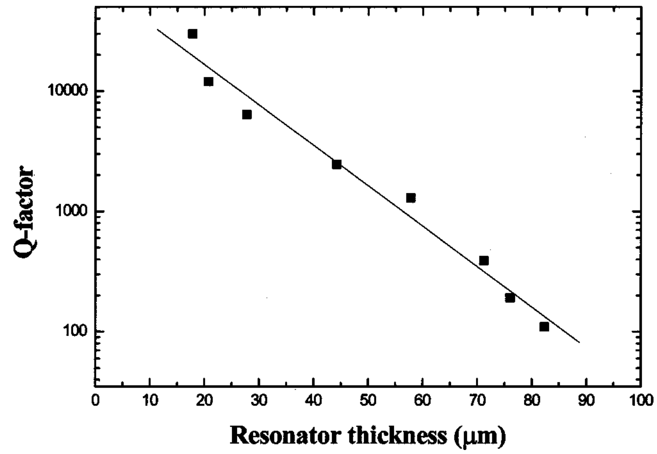

FIG. 3. Dependence of Q factor on the thickness of quartz resonators with diameter 1 mm.

quality of resonators. In our present work, the surface roughness of the etched region of the resonators was evaluated by a stylus profilometer and atomic force microscope measurement. The estimated average surface roughness of the etched resonators was found to be in the range 4–8 nm.

The electrical characteristics of the sensor were studied using a Hewlett Packard impedance/gain-phase analyzer (HP 4194 A). The resonance frequencies of quartz resonators were evaluated from the conductance (admittance magnitude) characteristics. All the measurements just mentioned have been carried out at room temperature.

We have studied the electrical characteristics of the fabricated quartz resonators with different thicknesses and diameters. It has been observed that, in the case of the resonator thickness ~82.3 μm and diameter 1 mm, the conductance versus frequency curve showed a fundamental resonance peak at 20.3 MHz and the Q factor was found to be ~100 [Fig. 2(a)]. However, the conductance versus frequency curve for the quartz resonator with the same diameter (1 mm) but with the thickness ~20.7 μm showed several resonance peaks [Fig. 2(b)]. This clearly indicates that, in the case of a thinner quartz resonator, spurious modes can be observed along with the fundamental mode. The appearance of several spurious modes might be due to the fact that, besides the geometric parameters of the electrodes, the possible formation of etch pits during the fabrication process also might have an influence. The Q factor of this resonator corresponding to the fundamental mode was found to be ~12 000. Figures 2(b) and 2(c) show the conductance versus frequency curves for the quartz resonators of the same thickness ~20.7 μm but with electrode diameters of 1 mm and 0.1 mm, respectively. The conductance spectra of the resonator with a diameter of 0.1 mm [Fig. 2(c)] shows fewer spurious peaks compared to that of 1 mm diameter resonator [Fig. 2(b)]. It is evident that reducing the electrode diameter (hence, the diameter of the resonator) results in an improvement in the separation of spurious modes from the fundamental vibration mode, which is in good agreement with the analysis reported in literature.[7] This, in turn, implies that cross talk between neighboring frequencies can be reduced by the miniaturization of the quartz resonators. The dependence of the Q factor on the thickness of quartz resonator is shown in Fig. 3. It can be seen that the Q factor increases with the reduction in

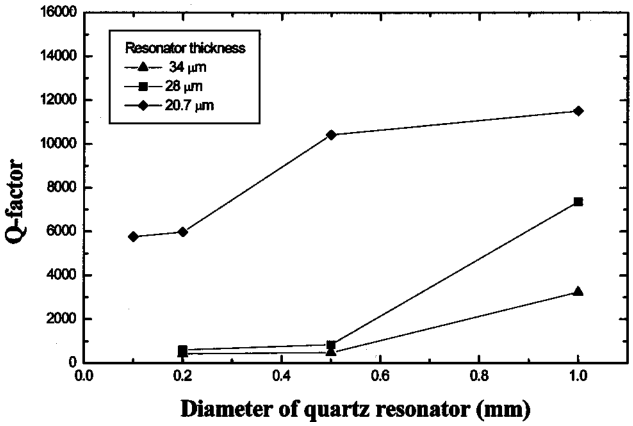

FIG. 4. Dependence of Q factor on the quartz resonator diameter.

thickness of the quartz resonator. For the quartz resonator with a thickness ~17.8 μm, corresponding to the fundamental resonance frequency 94 MHz, the Q factor reaches a value of 30 000. It is observed that the decrease of resonator thickness results in the increase of both resonance frequency as well as Q factor.

Figure 4 presents the dependence of the Q factor on the diameter of the resonator. It can be seen that the Q factor decreases with the reduction of the diameter of the resonator. However, as mentioned herein, for resonators of the same diameter, the Q factor increases with the reduction of the resonator thickness. Further, it is to be noted that it is possible to detect the fundamental resonance peak of the resonator with a thickness ~20.7 μm whose diameter is as small as 0.1 mm. Also, the Q-factor value for this is found to be 5700, which is not too low even though the resonator diameter is small.

In essence, in this letter, we are presenting the fabrication of a miniaturized highly sensitive single-chip multichannel QCM using the deep RIE technique. It is shown that thinning the quartz resonators improves their performance. The development of the miniaturized highly sensitive single-chip quartz resonator arrays opens up several application possibilities in areas such as microsystems and microreaction technology.

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